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This report outlines the design, construction, and implementation of a Nano-modulated ceramic coating deposition system. The deposition system was designed to deposit multilayer (superlattice) coatings of two or more oxide or other non-conducting materials. The system has two cathodes opposed to each other with their magnetic fields forming a closed loop, which enhances the current into the substrate. The deposition system takes advantage of the latest developments in vacuum and characterization technologies to ensure a repeatable process. It is equipped with cryopumps on the main chamber and the load lock for fast pumpdown and minimization of system contamination. Peripheral equipment includes an rf plasma probe for determination in real time of the plasma impedance, and an in-situ ellipsometer for real time characterization of one of the films making up the superlattice.

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Final Report

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TO

UNITED STATES AIR FORCE
AIR FORCE OFFICE OF SCIENTIFIC RESEARCH

FOR

A NANO-MODULATED CERAMIC COATING DEPOSITION SYSTEM

by

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October 31, 1996

1. INTRODUCTION

This is the final report on A Nano-Modulated Ceramic Coating Deposition System. This effort was funded by the United States Air Force Office of Scientific Research with a grant of $350,000 and a BIRL/Northwestern University contribution of $ 122,128, for a total of $472,128. The funding grant provided for the design and construction of a sputter deposition system capable of producing nano-layer composites of ceramic materials. The work on this system was started in August 1995. Delivery of the deposition chamber was in February 1996. Peripheral equipment, such as pumps, power supplies, and gauging was delivered such that the equipment would be available when needed.

The site for the system was prepared prior to the delivery of the chamber. Sight preparation consisted of providing sufficient water, electrical power, and air to operate the system. Delivery of the chamber was the first step in the construction of the deposition system. The various purchased items were installed onto the chamber and checked for functionality. This included the pumps for the chamber and load lock, the sputter sources, the power supplies and all of the associated measuring equipment.

The deposition system is now fully functional. Base pressures in the load lock and the deposition chamber itself are in the 10^{-7} Torr range. The chamber is fully instrumented, with the exception of the ellipsometer. The ellipsometer is still being used on the bench to measure coating optical properties after deposition. The ellipsometer will be installed and used as a process control once we are thoroughly familiar with its operation and capabilities.

Several graduate students from the Materials Science Department at Northwestern University are currently sharing the use of the deposition chamber for experimental work on their theses. This work is being supported by both Air Force (AFOSR) and industrial contracts.

1.1. Equipment Description

The deposition system is shown schematically in Figure 1. To avoid clutter, we have not included all of the peripheral equipment in the schematic diagram. The system is equipped with cryogenic high vacuum pumps on both the main chamber and the load lock. Both the main chamber and the load lock have base pressures in the low 10^{-7} Torr range. The load lock will achieve a vacuum level of approximately 1×10^{-5} from atmosphere within 5 minutes after initiating the pumping cycle. At this pressure level in the load lock, very little contamination is transferred into the deposition chamber. This means that the deposition chamber recovery time (the time required to achieve the desired base pressure after exposure to contamination) is very short, typically on the order of a few minutes. The single roughing pump on the system serves both to rough out the load lock and to rough out the deposition chamber when it is vented to atmosphere for target changes.

The sputtering sources are strongly unbalanced magnetrons arranged in a closed field configuration. This set up produces a maximum in ionization at the substrate. The cathodes are mounted through the doors of the deposition system, on umbilicals which can be adjusted to change the target to substrate distance. The cathodes are capable of accepting either

directly cooled targets or targets mounted to backing plates. This allows the use of a wider variety of target materials other than just metals, i. e., hot pressed materials can also be sputtered.

A substrate table has been designed and built. The table has a variable speed rotation capability and can be biased with either rf, dc or pulsed dc power. Designed into the table, but not yet built into it, are capabilities for thermocouple and Langmuir probe measurements. These capabilities will be added as required for the various programs that the deposition system will be used for. Also designed into the substrate table, but not yet implemented, is the capability to heat to temperatures suitable for epitaxial growth of some thin film materials.

Power to the cathodes is provided by 10 kW Advanced Energy power supplies equipped with SPARC-LE units which are capable of operation at frequencies up to 100 kHz. The frequency range is important because higher frequencies will prevent arcing from oxides, or other insulating materials, growing on the target surface. A 10 kW Advanced Energy power supply with a SPARC-LE unit has also been provided for substrate bias. The variable frequency SPARC-LE units can be synchronized together to minimize problems with plasma shutdowns when different frequencies or duty cycles are used. Presently, up to three SPARC-LE units can be synchronized together.

A bias frequency higher than that which can be supplied by the variable frequency SPARC-LE units will occasionally be required. We have therefore installed a 13.56 MHz rf bias capability at the substrate. We have also included in the design of the system an rf probe that will measure the real voltage, current, and impedance of the plasma near the substrate surface. Knowledge of the true values of these parameters will greatly assist in developing processes which require an rf bias.

The deposition system has been designed with the latest and best process controls available to us at the time of construction. We have installed a Spinning Rotor Gauge to measure true chamber pressure, a STABIL ™ ion gauge which, because it doesn't drift, will give an accurate chamber pressure measurement over a long period of time, a high accuracy capacitance manometer for measurement of true deposition pressures, a fast response mass spectrometer for control of the reactive gas partial pressure, and a modified gas flow control system with quick response piezoelectric valves to rapidly change the gas flow into the system as required by the deposition process.

A variable wavelength ellipsometer has been purchased to install on the deposition chamber as part of a control loop for control of coating properties. For in-situ use, this instrument will be set up to monitor only a single wavelength rather than the entire visible spectrum. This will insure that the response time of the instrument is such that corrections to relevant deposition parameters can be made in order to keep the deposition process on track. Currently, this instrument is being used on the bench to characterize the optical properties of the coatings being deposited.

Substrate heat has been provided for, but not yet installed into the deposition chamber. Heat will be installed when required by the coating work being conducted in the chamber. The heater should be capable of temperatures up to approximately 400 degrees Celsius.

A computer has been designated for use with the deposition system. Hardware for deposition process control has been purchased, and we are currently writing the software required for its use. When complete, the system will be capable of operation either manually or under computer control.

1.2. Equipment purchases

The various items of equipment, purchased with AFOSR funds, to construct this deposition system are listed in Table 1, along with their original estimated costs. A lsit of purchase orders issued for this project and their costs is shown in Table 2. Deviations from the originally planned purchases are discussed in the following section of this report. Some deviations were required because the design of the system had not been finalized at the time of the award of the grant, or new equipment had become available that wasn't on the market at the time the proposal was submitted. The final design was part of the Northwestern contribution to the overall program.

Table 3 lists BIRL's originally planned contributions to the overall effort. Table 4 lists AFOSR's and BIRL's contributions to the labor required to construct the deposition system from the purchased parts. The Air Force's contributions were limited to the $ 350,000 appropriation, while BIRL's contributions exceeded the estimated $122,128 by a significant amount.

Table 1. Equipment purchased with AFOSR funds for construction of the Nano-Modulated Ceramic Sputter Deposition System.

Original Plan			Actual Purchases
Item	Original Vendor	Estimated Cost k	Vendor[1]
Deposition Chamber	Meyer Tool	37	Meyer Tool
Load Lock	"	12	"
Cryopump	CTI	8	CTI
Sputter Power Supplies	Advanced Energy	40	Advanced Energy
SPARC-LE Units	Advanced Energy	12	Advanced Energy
RF Plasma Probe	"	7	"
Sputter Sources	Materials Science Inc.	20	Gencoa Ltd.
Mass Spectro-meter	MKS Instruments	20	Spectra
Gas Flow Control	MKS Instruments	15	MKS Instruments
Roughing Line Hardware	Various	6.0	Various-MKS Various -MDC Various - Lesker
Throttle Valve	"	5	MKS Instruments
Mechanical Pump	Leybold-Heraeus	4	Alcatel
STABIL-ion Gauge	Granville Phillips	4	Granville Phillips
Gate Valves	Midwest Vacuum	12	Midwest Vacuum
Feed-throughs	Various	8	Various[2]-Lesker -MDC -MKS
Substrate Heater	Denton Vacuum	5	CHA Industries
Computer	Local Vendor	3	BIRL Contribution
Control Software	National Instruments	2	National Instruments
Substrate Holder Components	Various	3	Various-Meyer Tool, -NU machine shop
Ellipsometer	JA Woollam Co.	54	JA Woollam Co.
Miscellaneous	Various Vendors	20	Various Vendors
Amount Requested		$297,000	
Amount spent		$299,000	

1. Vendors full names and addresses are given in Appendix 1.

Table 2. Purchase orders, vendors and costs for equipment purchased for construction of the ceramic coating deposition system

PO Number	Vendor	Description	Cost $
RX001039	Meyer Tool	Chamber	41,000
RX001041	MKS Instr.	Flow Cont.	10,457
RX001043	Spectra	Mass Spectr.	18,500
1044	Gencoa Ltd.	Sputter Sources	19,600
1046	Meyer Tool	Load Lock	10,900
1052	CTI	Cryopump	7,995
1054	VAT	Gate Valve	6,795
1055	MDC Vac. Prod.	Adapter	5,451
1091	Adv. Energy	RF Power Supply	13,585
1093	JA Woollam	Ellipsometer	54,000
1111	Adv. Energy	SPARC-LEs	12,000
PD003589	Granville Phillips	STABIL ion gauge	4,100
3590	Alcatel	Pump	3,032
3607	MKS Instr.	Capacitance Manometer	3,660
3608	MKS	Throttle Valve	4,626
3634	VAT	Gate Valve	4,185
3690	Maxtek	Piezo valves	1,290
3691	Austin Scientific	Cryo absorber	450
3692	Copper and Brass Sales	Hard Copper roll	529
3696	Austin Scientific	Cryo fittings	452
3701	E Besler	Import fees	799
3738	MKS	Vacuum fittings	3,529
3748	Kurt J. Lesker	Vac. Fittings, Feedthroughs	4,734
3751	Nor-Cal Products	Foreline Trap	8,985
3774	CHA Industries	Heater Pwr Supply	4,007
3804	Adv. Energy	Power Supplies	37,791
3815	Meyer Tool	Frame Modification	2,490
3923	MKS	Spinning Rotor Gauge	6,500
3966	MKS	Vac. Fittings	1,039
3967	Newark Electronics	Elec. Comp.	240
3983	McMaster Carr	Misc.	327
4020	Instr. Assoc.	Misc.	425
4077	Newark	Misc. Elec.	1,047
4078	Evergreen Oak	Wire	332
4162	Ash Equip.	Heater	274
4169	Natl. Instr.	Computer Hdwr.	1,458
4338	Ash Equip.	Heater	270
4355	Instr. Assoc.	Tube Fittings	561
4374	Grainger	Fittings	59
4379	McMaster Carr	Tube Fittings	189
4495	Meyer Tool	Subst. Fixture	1,830
4508	Lesker	Flow Switches	390

4511	McMaster Carr	Tubing	57
4512	Dearborn Valve	Valves	374
4513	Bradley Supply	Tube Fittings	228
4515	Instr. Assoc.	Connectors	626
4551	Bradley Supply	Connectors	54
4569	Berry Bearing	Bearings	131
4570	Stock Drive Prod.	Sprockets	144
4586	Grainger	Gear Motors	642
4588	Bradley Supply	Ball Valves	52
4600	Newark	Misc. Elec.	44
4608	Instr. Assoc.	Connectors	177
4667	Dearborn Valve	Connectors	38
4668	Instr. Assoc.	Fittings	19
4669	Nor-Cal Prod	Adapter	1,660
4700	Instr. Assoc.	Connectors	72
4743	McMaster-Carr	Tubing, Misc.	196
4752	Dearborn Valve	Misc. Fittings	138
4753	Instr. Assoc.	Misc. Fittings	110
4774	Meyer Tool	Flange Assy.	1,580
4799	McMaster-Carr	Al. Rod	93
4800	McMaster-Carr	Misc. Parts	85
4814	Revere Electric	Conduit	53
4836	Granville Philips	Spare Parts	805

Table 3. BIRL's Equipment Contributions

Original Plan		Actual Vendor Cost
Description	**Estimated Value K**	
Frame and Instr. Racks	4	BIRL Provided
Utilities	8	Contractors
Load Lock Cryopump	8	BIRL Provided
Valve Sequencers	5	BIRL Provided
Process Control Panel	5	BIRL Provided
Laboratory Modifications	10	BIRL Provided
Deposition System Design	11	BIRL Provided
Spinning Rotor Gauge	12	MKS Instruments
Ion Gauge (LL)	2	BIRL Provided
RF Power Supply	10	Advanced Energy
Total Estimated	$75,000	

Table 4. AFOSR and BIRL labor dollars

AFOSR		BIRL	
Appropriated	Spent	Initial Estimate	Spent
$53,000	$51,000	$47,145	$96,000

Special Circumstances of Acquisition: None

Changes in Original Equipment list:

Additional Items:

1. Mass Spectrometer: Purchased from Spectra rather than MKS because of better response time and better price.

2. SPARC-LE Units: Variable frequency units became available between the time the proposal was written and the grant award. The variable frequency units are more expensive than those originally allowed for. Therefore, two of the variable frequency units were purchased with AFOSR grant money and one was purchased by BIRL.

3. RF plasma probe: This device was originally scheduled to be purchased with AFOSR grant money. Instead, the vendor, Advanced Energy, has provided a probe on an evaluation basis.

4. Sputter Sources: A vendor other than the one shown in the original proposal was chosen as the supplier of the sputter sources. The sputter sources were purchased form Gencoa because of their experience with the fabrication of unbalanced magnetrons.

5. Rough Pump: Alcatel was chosen instead of Leybold-Heraeus because of price.

6. Substrate Heater: The substrate heater power supply was purchased from CHA instead of Denton. This was done because of price.

7. Computer: BIRL contributed a computer instead of purchasing on using grant funds.

8. RF Power Supply: This item was originally to be contributed by BIRL. However, it was purchased using AFOSR funds. Additional labor dollars were contributed by BIRL to compensate for the purchase.

Several additional purchases, not itemized in the proposal, were made. These were necessitated by system design changes made after the award of the grant. As examples, several adapters were required to mate the gate valves purchased to the final chamber design.

Deleted Items: There were several changes from the original list of items to be purchased or provided. However, no items were completely eliminated.

1.3. Summary of Research Projects

The deposition system is quite new and to date we have had time to use it for only one research project, which is being sponsored by AFOSR. The project entitled "Synthesis and Characterization of Oxide Superlattice Coatings" and is Grant No. F49620-95-1-0177. the scope of the work being done on this project is to use high rate reactive dc magnetron sputtering to deposit superlattice films of oxide materials. These materials are expected to have enhanced mechanical properties, such as hardness and abrasion resistance, optical properties that can be controlled by controlling ratio of the materials used in a superlattice period, and are also expected to have excellent thermal barrier properties.

Appendix

Vendors Used

Chamber/Load Lock	Meyer Tool 4601 Southwest Highway Oak Lawn, Illinois 60453 (708) 425-9080
Cryopump	CTI Technical Engineering Associates 7920 Lakeville Blvd. Lakeville, MN 55044 (612) 469-6500
Sputter Power Supplies	Advanced Energy Industries Technical Engineering Associates (see above)
SPARC-LE Units	Advanced Energy Industries Technical Engineering Associates (see above)
RF Plasma Probe	Advanced Energy Industries 1625 Sharp Point Drive Fort Collins, CO 80525 (970) 221-4670
Sputter Sources	Gencoa LTD. 4 Wavertree Boulevard South Wavertree Technology Park Liverpool, L7 9PF, UK 44-151-252-2200
Mass Spectrometer	Spectra 700 A East Dunne Ave. Morgan Hill, CA 95037 (408) 778-6060
Gas Flow Control	MKS Instruments 13341 Southwest Highway Orland Park, IL 60462 (708) 923-0222
Roughing Line Hardware	1. MKS Instruments (see above)

	2. MDC Vacuum Products Advent Associates 5808 N. St. Louis Ave. Chicago, IL 60659 (773) 583-7979
	3. Kurt J. Lesker 1515 Worthington Ave. Clairton, PA 15025 1-800-245-1656
Throttle Valve Mechanical Pump	MKS Instruments (see above) Alcatel Midwest Vacuum 201 E. Ogden Ave. Suite 15-1 Hinsdale, IL 60521 (630) 323-5399
STABIL Ion Gauge	Granville Phillips 5675 Arapahoe Ave. Boulder, CO 80303 1-800-776-6543
Feedthroughs	Kurt J. Lesker (see above) MKS Instruments (see above) MDC Vacuum Products (see above)
Substrate Heater	CHA Industries Byron Ellis Associates 650 First Ave. Des Plaines, IL 60016 (847) 298-7626
Gate Valves	VAT Midwest Vacuum (see above)
Computer	BIRL contribution
Control Software	National Instruments 6504 Bridge Point Parkway Austin, Texas 78730-5039 1-800-433-3844
Substrate Holder Components	1. Meyer Tool (see above) 2. NU machine shop

3. Bradley Supply Co.
   PO Box 29096
   Chicago, IL 60629
   (312) 434-7400

4. Instrument Associates
   4833 W 128 St.
   Alsip, IL 60658
   (708) 597-9880

5. Grainger
   8045 River Drive
   Morton Grove, IL 60053
   (847) 965-7600

Ellipsometer

JA Woollam Co.
650 J Street
Lincoln, NE 68508
402-477-7501

Miscellaneous

1. Dearborn Valve and Fitting
   PO Box 847
   Wauconda, IL 60084
   (630) 526-6800

2. Newark Electronics
   1365 Wiley Road
   Schaumberg, IL 60173
   (847) 310-8980

3. Revere Electric Supply
   617 Church Road
   Elgin, IL 60123
   (630) 741-8900

4. MAXTEK INC.
   2908 Oregon court
   Torrance, CA 90503
   (310) 320-6604

5. E Besler & Co.
   115 Martin Lane
   Elk Grove Village, IL 60007
   (630) 364-0300

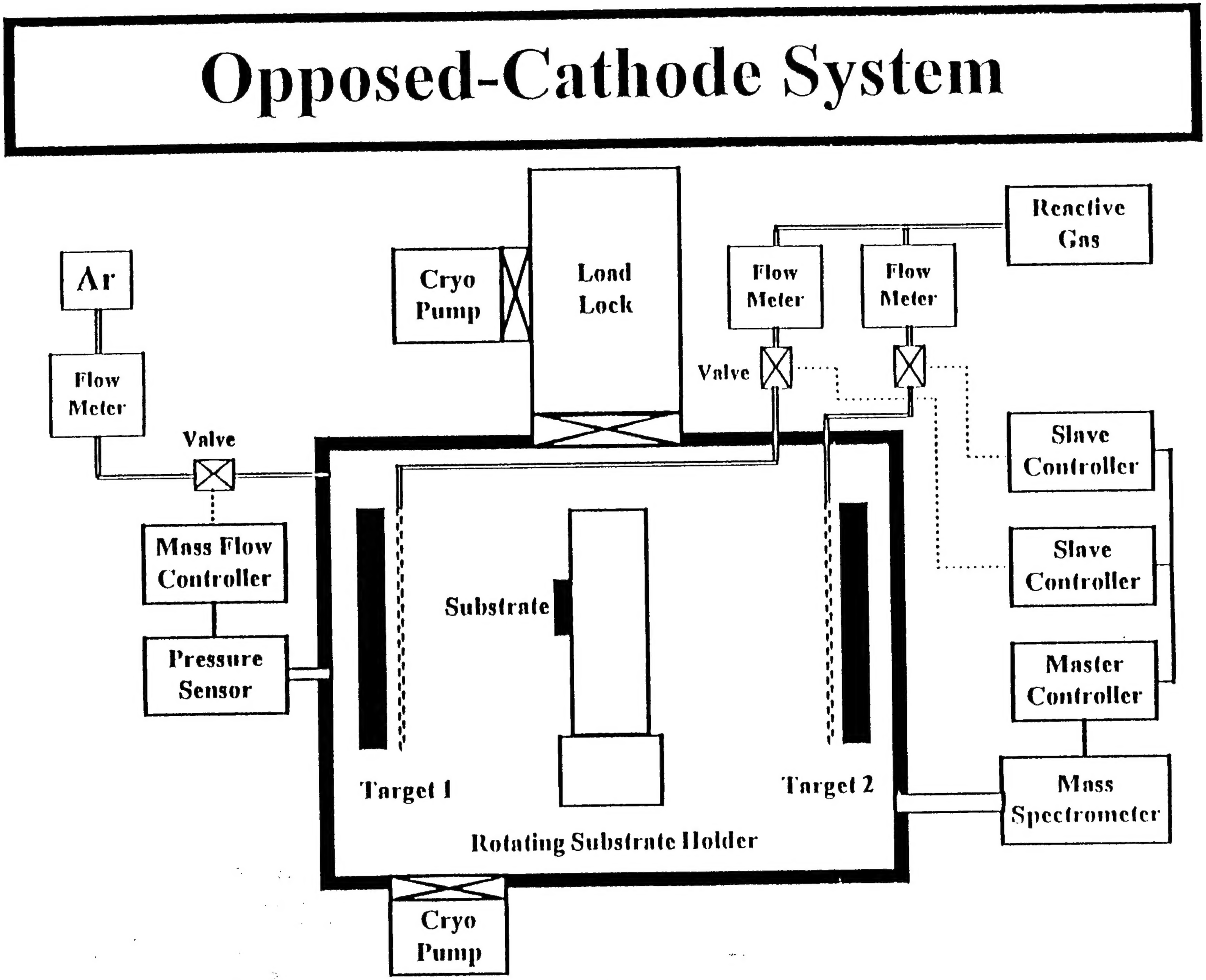

Figure 1. Schematic representation of the ceramic coating deposition system.